



Three electrolyte high voltage acid–alkaline hybrid rechargeable battery

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ABSTRACT

Performance characteristics of a three electrolyte rechargeable acid–alkaline hybrid battery using a PbO_2 positive plate and a nickel metal hydride (NiMH_x) negative electrode in separate electrolyte of H_2SO_4 and KOH were studied. This hybrid battery has three electrolytes in a single cell. A neutral K_2SO_4 salt solution was placed between the acid and alkaline compartments of the cell, in which a cation exchange membrane and an anion exchange membrane, were employed to separate these three electrolytes. The open circuit voltage of this hybrid cell was found to be 2.64 V in an electrolyte configuration of 1 M H_2SO_4 |0.2 M K_2SO_4 |2 M KOH electrolyte configuration, compared to 1.92 V in the conventional lead–acid cell in 1 M H_2SO_4 and 1.40 V in a NiMH_x cell in 2 M KOH . This hybrid acid–alkaline $\text{PbO}_2/\text{NiMH}_x$ battery was shown to operate with a voltage 20% higher than the conventional lead acid battery and 110% higher than nickel–metal hydride battery at 1/3 C discharging rate. The concentrations of the three electrolytes, the dimension of the electrolyte chamber, and other cell/operation parameters with impacts on the hybrid cell performance were investigated.

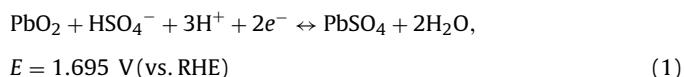
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1. Introduction

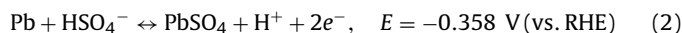
With expanded application of electric power in households and industries, the demand for lower-cost efficient power sources is strong with primary and rechargeable batteries employed in a large number of applications [1]. The operating voltage of aqueous power sources is limited by the electrochemical window of water which has a theoretical value of 1.229 V. Outside this window, oxygen or hydrogen evolution may occur. Due to the electrochemistry of the $\text{PbO}_2/\text{PbSO}_4/\text{H}_2\text{SO}_4$ system, the OCV of lead acid battery can be as high as 2.05 V, which is the highest among aqueous batteries.

For aqueous power sources, most electrode reaction potentials shift with pH variation according to the Nernst equation. This can be demonstrated by the Pourbaix diagram for Pb species [2] in Fig. 1. The electrode potential of PbO_2 or Pb has a linear relationship with the pH of the solution. The open circuit voltage (OCV) and working voltage change with pH since both positive and negative electrode reactions involve generation or consumption of protons.

The electrode reactions of lead acid battery are [2,3]:

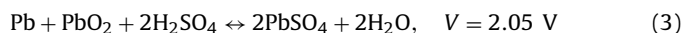


at the positive electrode; and



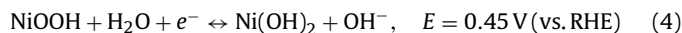
at the negative electrode.

Thus, the overall electrode reaction is

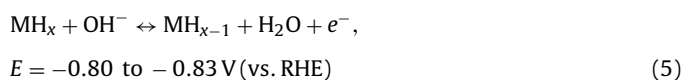


As shown in Eq. (3), H_2SO_4 is an active component affecting lead acid battery performance. Applying the Nernst equation to the lead–acid battery, a simplified equation $V_{\text{OCV}} = S.G._{\text{H}_2\text{SO}_4}(\text{specific gravity of } \text{H}_2\text{SO}_4) + 0.85$ is commonly employed to describe the effect of H_2SO_4 concentration on the OCV of a lead acid battery. This indicates that the lead acid battery can be operated with a higher voltage in a more acidic electrolyte.

Similar phenomenon could be applied to nickel metal hydride (Ni–MH_x) battery in alkaline solution. The half cell reactions on the positive and negative electrodes of NiMH_x cell during discharging/charging process are [4].



at the positive electrode; and



at the negative electrode.

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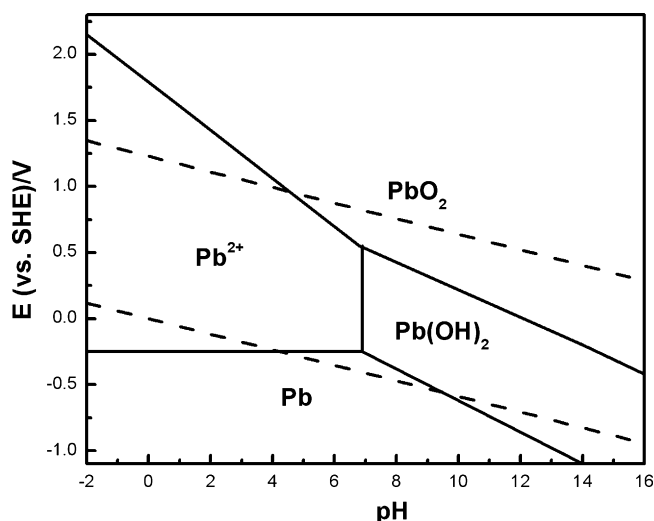
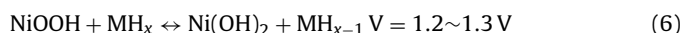


Fig. 1. Pourbaix diagram of Pb species [2].

The overall electrode reaction is



Our previous work has shown that a much higher cell voltage is obtained when reaction (1) in an acidic electrolyte is coupled with reaction (5) in an alkaline electrolyte [5–7]. A MB-3 bipolar membrane from Membrane Technology Centre (Russia) was employed in the middle of the battery compartment to separate the acid and alkaline electrolytes to avoid the self-neutralization. The open circuit voltage (OCV) of such a hybrid battery was higher, at about 2.60 V at room temperature in the 5 M H_2SO_4 /7 M KOH dual-electrolyte system [5–7]. Though the results were impressive, these bipolar membranes are expensive and difficult to obtain in large quantities. More importantly, the bipolar membrane cannot operate with a current density larger than 20–50 mA/cm², limiting its wider application as battery [8–15]. Other researchers reported construction of acid–alkaline hybrid power sources like acid–alkaline hybrid fuel cell and acid–alkaline battery [16–20]. Kohl and coworkers reported a hybrid membrane-electrode assembly (MEA) composed of acidic and alkaline solid electrolytes forming essentially a bipolar membrane interface [17,18]. Their hybrid MEA offered better water management and higher steady-state current in a hydrogen–oxygen fuel cell. Wang et al. [19] reported an acid–alkaline hybrid direct borohydride fuel cells (DBFCs) with a ceramic lithium super-ionic conductor (LISICON) film. The LISICON film based acid–alkaline hybrid DBPFC could provide a high cell voltage (2.1 V), but its discharging performance is greatly limited by the ionic conductivity of the LISICON film with a maximal power density at 1.54 mW cm^{−2}. Zholkovskij et al. [20] explored the applicability of bipolar membranes in storage batteries, but concluded that an industrial application for such batteries is barely achievable due to the low energy density of 0.5 W/kg. In this study, we put forward an alternative configuration of ionic interface that allows both acid and alkaline solutions co-existing in a single hybrid battery system but without a bipolar membrane. An additional gap/compartment filled with neutral salt of K_2SO_4 is created between the cation exchange membrane (CEM) and anion exchange membrane (AEM) to separate the respective acid and alkaline solution compartments. The costs of a pair of CEM and AEM are lower than that of a bipolar membrane, which is less readily available commercially. As shown in Fig. 2, PbO_2 positive electrode was placed in the H_2SO_4 electrolyte, which is separated from the K_2SO_4 solution by an AEM, and NiMH_x negative electrode in KOH electrolyte is separated from the K_2SO_4 solution by a CEM.

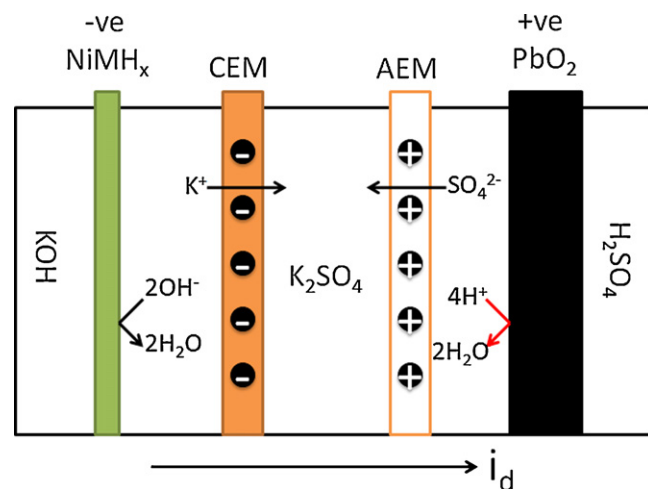
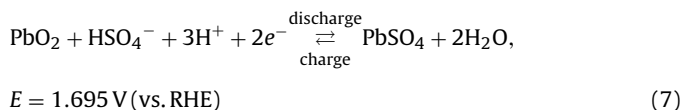
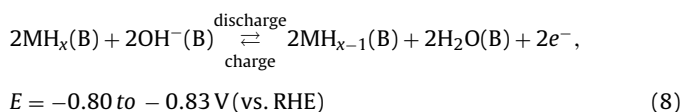


Fig. 2. Three compartment cell arrangement of hybrid acid–alkaline battery and the possible current flow directions during charging and discharging processes.

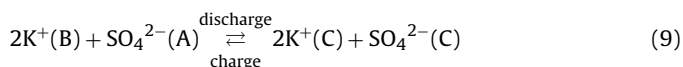
During the discharging (or charging) process, the electrode reactions on positive and negative electrodes are



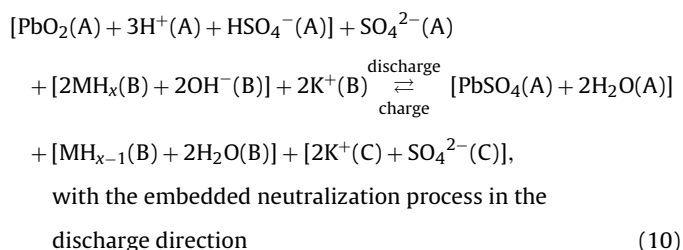
on the positive electrode in acidic solution; and



on the negative electrode in alkaline solution; while the reaction in the neutral solution of the central compartment:



The overall cell reaction is:



This process may be termed electrochemical neutralization since the protons and hydroxide ions are converted to water in separate chambers with corresponding charge and species balance and the pH difference created the extra free energy and voltage. The ΔG of the neutralization reaction in Eq. (10) can be calculated to be -79.9 kJ per mole of water formed, corresponding to 0.828 V according to Nernst equation. How much of this additional voltage or free energy be realized depends on other free energy losses including the transfer of ions across membranes as in Eq. (9).

Here the items of A, B and C in brackets denotes the material in acidic, alkaline and central salt chambers respectively. Due to the selectivity of CEM and AEM membranes, most ions or compound cannot be transferred from one chamber to another chamber. The ionic reactions in this three-electrolyte hybrid battery are different from the dual electrolyte battery with a bipolar membrane due to

different current carriers during the charging or discharging process. In the dual electrolyte system, H^+ and OH^- are the only charge carriers. In three electrolyte configuration, the K^+ and SO_4^{2-} move into the central compartment, from the alkaline and acid chambers, respectively, to maintain charge balance and function as current carriers during discharge, as shown in Eq. (10). Fundamentally, the three-electrolyte configuration provides alternative ionic interface without the need of water splitting reaction.

During discharge (as illustrated by Eqs. (7) and (8)), H^+ (A) and OH^- (B) ions are consumed on the positive and negative electrode, respectively. There will be an excess of SO_4^{2-} and K^+ ions in the acid and alkaline chambers. With the discharge current flowing through the circuit, the excess SO_4^{2-} and K^+ ions must transport into the central compartment to maintain the charge balance of each chamber (Eq. (9)). During charging, excess H^+ (A) and OH^- (B) ions are produced on the positive and negative electrode, respectively. The salt ions in the centre chamber will transport into the acid and alkaline compartments to maintain the charge balance and function as the current carrier in the electrolytes. Therefore, a ions combination process occurred in the central compartment, in which the free energy of the corresponding neutralization is transformed into electrochemical energy (Eq. (10)). According to the Nernst equation, the -79.9 kJ/mol of water formation gives rise to the additional theoretical voltage of 0.83 V. But only a fraction of this additional voltage is realized due to transport resistance across the ionic interfaces. The OCV of this hybrid cell was observed to be 2.495 V.

The aim of this work is to evaluate the performance of a three electrolyte hybrid battery with PbO_2 positive electrode in H_2SO_4 solution and $NiMH_x$ negative plate in KOH electrolyte. A K_2SO_4 solution was sandwiched between the acid chamber and the alkaline chamber, separated by an AEM and a CEM, respectively. Factors including the electrolyte concentration and cell gap that affect the performance of this hybrid acid–alkaline $PbO_2/NiMH_x$ battery are also investigated.

2. Experimental

2.1. Three-electrolyte cell design

The hybrid battery had three compartments. The acid chamber contained a PbO_2 positive plate immersed in 1 M H_2SO_4 solution, while the alkaline chamber contained a MH_x negative plate immersed in 2 M KOH electrolyte. A separate chamber filled with a salt solution of 0.2 M K_2SO_4 was placed in the middle between the alkaline and acid chambers. The three chambers were separated by AEM and CEM with continuous ionic transport ensured.

The positive PbO_2 electrode was extracted from a commercial Yuasa NP10-6 lead-acid battery and had an active nominal surface area of 20 cm² with a mass of 49.9 g. The negative MH_x electrode was prepared by 2.8 g $LaNi_5$ based metal–hydride alloy powder extracted from a commercial GP-2700 AA battery and pressed onto one side of a nickel foam. The MH_x negative electrode had a total mass of 4.1 g and an approximate active nominal area of around 12 cm².

A cation exchange membrane (CEM) (CMI-7000S, Membrane International Inc., USA), and an anion exchange membrane (AEM) (AMI-7001S, Membrane International Inc., USA) were placed between the acidic and alkaline chambers and the central chamber. The perm-selectivity of CEM and AEM was tested with 0.1 mol/kg KCl and 0.5 mol/kg KCl solutions, to be 90% and 94% , respectively. The nominal area of each AEM and CEM is 24 cm² with dimensions of 40 mm (L) $\times$ 60 mm (H).

The volume of both acid and alkaline electrolyte is about 40 mL in a chamber of dimensions 46 mm (L) $\times$ 68 mm (H) $\times$ 18 mm

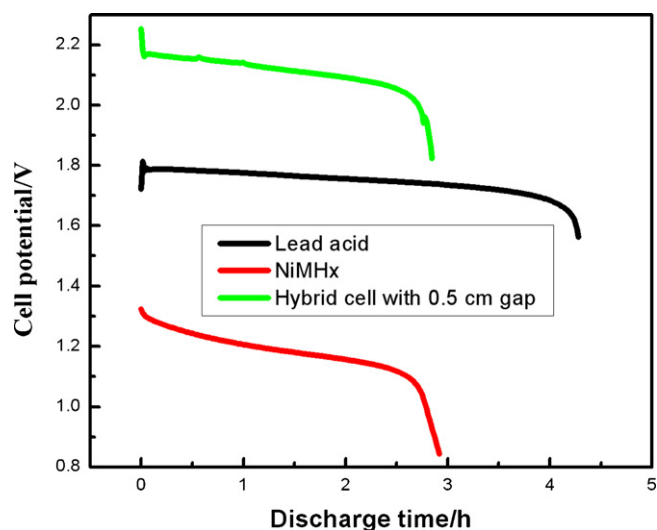


Fig. 3. Discharge performance of the hybrid acid–alkaline cell. The distance between AEM and CEM are fixed at 0.5 cm and the electrolytes are 1 M H_2SO_4 – 0.2 M K_2SO_4 – 2 M KOH . The discharging current was 100 mA. Performances of conventional lead acid battery using 1 M H_2SO_4 and conventional $NiMH_x$ battery using 2 M KOH is also included for comparison.

(W). The dimensions of the middle cavity are 46 mm (L) $\times$ 68 mm (H) $\times$ 5 mm (W) with 12 mL salt solution inside.

2.2. Electrochemical tests

All cell performance tests were carried out at room temperature. Galvanostatic charge/discharge measurements, and AC impedance tests were carried out with an Autolab PGSTA30 Potentiostat. The discharge current was kept at 100 mA in most runs. The frequency in the AC impedance experiments was ranged from 100 kHz to 10 mHz with 10 mV amplitude perturbation around the OCV. Hg_2SO_4/Hg and HgO/Hg are employed as reference electrodes in acid and alkaline chambers, respectively. The cyclic performance of the hybrid cell with 1 M H_2SO_4 – 0.2 M K_2SO_4 – 2 M KOH electrolyte was explored. Constant current cycling was performed with 3 -h charging followed by 3 -h discharge. The pre-set 3 -h charging or discharge is over-ride and shortened when charging exceeds 2.85 or discharge falls below 2.0 V.

3. Results and discussion

3.1. Cell performance of PbO_2/MH_x hybrid cell

Fig. 3 compares the discharging performance of the PbO_2/MH_x hybrid battery, with individual 1 M H_2SO_4 single electrolyte lead acid battery and 2 M KOH $NiMH_x$ single electrolyte battery. The hybrid battery consisted of the 1 M H_2SO_4 | 0.2 M K_2SO_4 | 2 M KOH electrolytes in the acidic, central and alkaline chambers, respectively. The discharging current for all systems are fixed at 100 mA but the cutoff voltage is different for each battery system. The cut-off voltage of the hybrid cell is fixed at 1.8 V, while that of lead acid battery is 1.5 V and 0.8 V for $NiMH_x$ battery. For the hybrid cell, the distance between AEM and CEM was fixed at 0.5 cm. In a typical cycle, it can be observed from Fig. 3 that hybrid cell had higher voltage discharge than that of individual lead acid and $NiMH_x$ batteries. The hybrid cell had a large OCV of around 2.60 V, which is significantly higher than that of individual lead acid (2.05 V) and $NiMH_x$ (1.35 V) batteries with the same electrolyte concentration. Based on the discharge curves of individual lead acid and $NiMH_x$ batteries in Fig. 3, the positive electrode was estimated to have a capacity of 400 mAh while the negative electrode had a capacity of

280 mAh when discharged at 100 mA. The hybrid cell was found to have an estimated capacity of 300 mAh, limited by the capacity of the NiMH_x electrode.

3.2. Cyclic performance of hybrid battery

Fig. 4 shows seven cycles of hybrid cell under a charge and/or discharge current of 100 mA. The charge/discharge process for the 5 cycles were fairly identical, the discharge voltage was able to maintain above 2.1 V over the 3 h duration. However, as the cycle increased, there was accumulation of salt in the middle salt chamber, this affected the discharge time slightly in the subsequent cycles.

3.3. Effect of the salt solution on the hybrid cell discharge performance

For an ionic solution, conductivity increases linearly with the concentration of the ions in the solution. Thus, the higher the salt concentration in the middle compartment, the lower the internal resistance of the hybrid cell. Fig. 5(a) compares the discharge in different concentrations of salt solution in the middle chamber whereas the concentration of the acidic and alkaline chambers remained at 1 M H_2SO_4 and 2 M KOH, respectively. As expected,

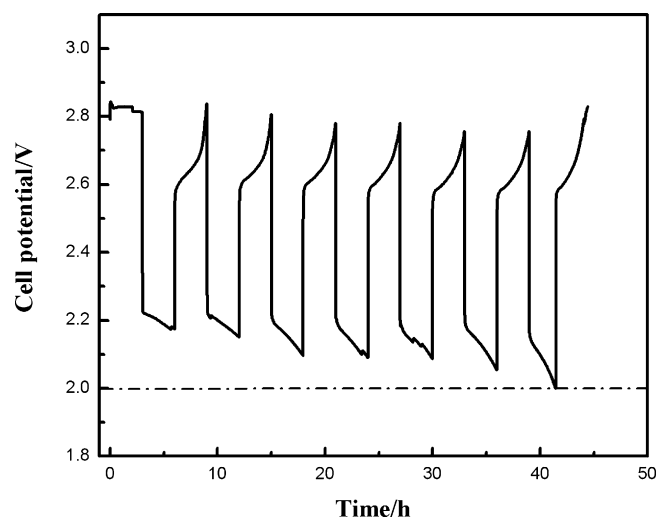


Fig. 4. Forty-hour charge–discharge cycles of $\text{PbO}_2\text{-MH}_x$ hybrid battery. The electrolytes are in 1 M H_2SO_4 |0.2 M K_2SO_4 |2 M KOH electrolyte configuration. The inter-membrane distance is 0.5 cm and the charging and discharging current is 100 mA.

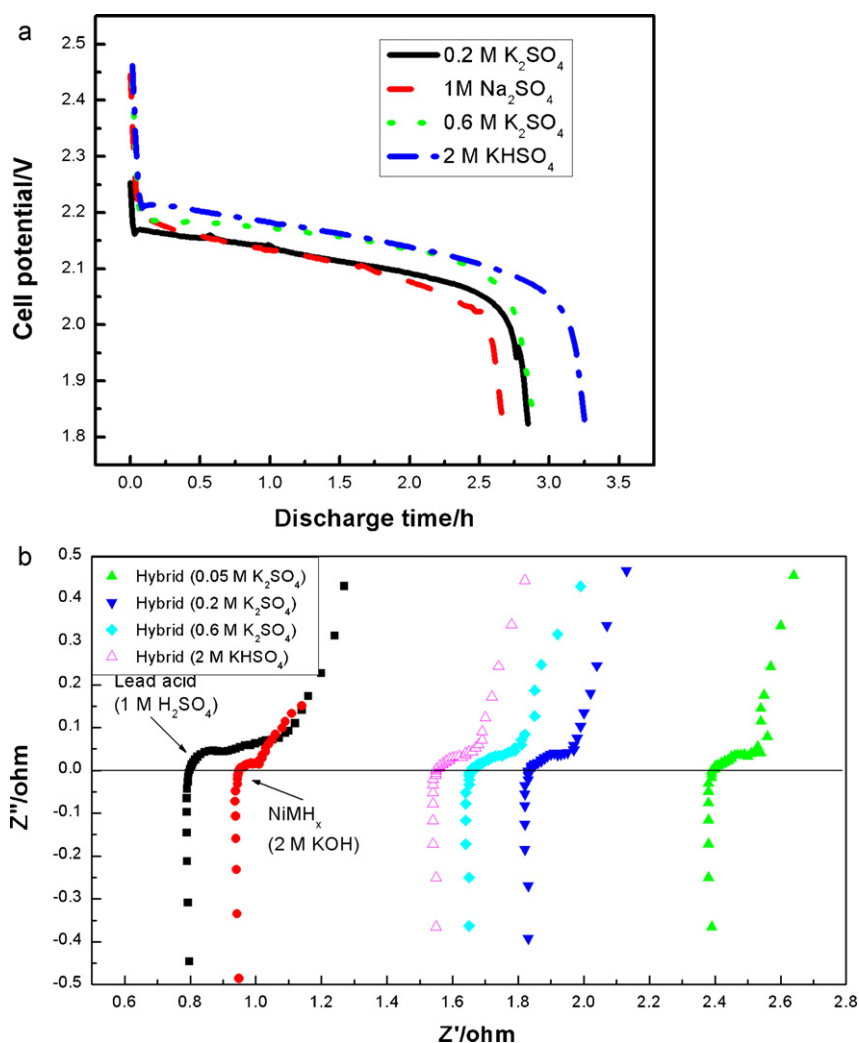


Fig. 5. (a) Discharge performance of hybrid cell with different salt solution concentrations in the middle compartment. The distance between CEM and AEM was fixed at 0.5 cm and the concentration of acid and alkaline was 1 M H_2SO_4 and 2 M KOH. The discharging current was 100 mA. (b) AC impedance test under OCV condition on the individual NiMH_x (in 2 M KOH) and lead acid batteries (in 1 M H_2SO_4), and that of the hybrid batteries in different neutral salt concentration at the central compartment.

the discharge capacity of hybrid cell increased as the concentration of the K_2SO_4 salt solution increased from 0.2 M to 0.6 M. The higher salt concentration, the higher conductivity and thus result in lower internal resistance. The performance of this hybrid cell was limited by the saturation concentration of K_2SO_4 which is only 0.63 M in water at room temperature. This saturation problem can be overcome by using an appropriate salt with higher solubility.

Comparing to K_2SO_4 , the solubility of KHSO_4 (3.57 M at 298 K) and Na_2SO_4 (1.37 M at 298 K) are much higher [21]. Hence these salts can be used as substitutes for K_2SO_4 . Fig. 5(a) also included the cell performances of hybrid cell with the use of 2 M KHSO_4 and 1 M Na_2SO_4 in the middle compartment with 2 M NaOH as the corresponding alkaline electrolyte. Hybrid cell with 1 M Na_2SO_4 had better performance in the initial discharging stage, but gradually the performance decayed to a level similar to that of 0.2 M K_2SO_4 salt. Solution conductivity test of these salt solutions shows that the conductivity of 1 M Na_2SO_4 was close to that of 0.6 M K_2SO_4 . Na^+ ions have lower limiting ion conductance ($50.20 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$) than that of K^+ ions ($73.55 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$) at 298 K in water [22], thus the conductivity of Na_2SO_4 is lower than that of equimolar K_2SO_4 solution. As shown in Fig. 5(a), having 2 M KHSO_4 as the neutral salt resulted in higher plateau voltage at 2.1 V and longer discharge time (3 h). This improved performance could be attributed to the low perm-selectivity of CEM as a result of large amounts OH^- being diffused from the alkaline chamber into the middle chamber. The OH^- ions then reacted with KHSO_4 to form K_2SO_4 . When K_2SO_4 concentration reaches the saturation limit, salt will be formed and occasionally observed on the surface of the membranes after several charging–discharging cycles. Salting out is a severe problem for the three electrolyte hybrid cell due to the inherent limitation of solubility of the salt electrolyte in the central chamber.

AC impedance test under OCV condition was carried out to estimate the internal resistance of the individual NiMH_x (in 2 M KOH) and lead acid batteries (in 1 H_2SO_4), and that of the hybrid batteries at various salt concentration of the central compartment. As illustrated by comparing the x-intercepts in Fig. 5(b), lead acid battery had the smallest internal resistance of 0.79Ω , while the hybrid cell had the largest internal resistance among these cells. The large internal resistance of hybrid cell could be attributed to the membrane resistance and the additional distance between the positive and negative plates in the three-compartment configuration.

3.4. Effect of concentrations in the acid and alkaline chambers

Apart from the salt solution in the middle compartment, the acid and alkaline electrolyte concentrations would also affect the hybrid cell performance. Fig. 6 compares the cell performance of various concentrations of acid and alkaline chambers (1 M H_2SO_4 |2 M KOH, 2 M H_2SO_4 |4 M KOH and 5 M H_2SO_4 |10 M KOH while the neutral salt is kept at 0.2 M K_2SO_4 . With increasing acid–alkaline concentration combination, the hybrid cell can be charged up to 3.0 V (with the 5 M H_2SO_4 |10 M KOH combinations) and discharged for 3 h to 1.8 V. Moreover, the discharge plateau remained as 2.3 V for 5 M H_2SO_4 |10 M KOH combination, compared to only 2.1 and 2.15 V for the lower acid–base combination of 1 M H_2SO_4 |2 M KOH and 2 M H_2SO_4 |4 M KOH, respectively. This was mainly due to the better electrode performance of individual positive and negative plate in concentrated acid and alkaline solution, as shown in Fig. 7. Fig. 7 revealed that the positive potential increased linearly with increasing acid concentration according to Eq. (1), while that of the negative potential was decreased negatively in a linear scale due to the higher alkaline concentration, according to Eq. (5). Thus the potential difference between two electrodes could be increased by the higher concentration electrolytes. However, higher concentration leads to higher concentration gradient across

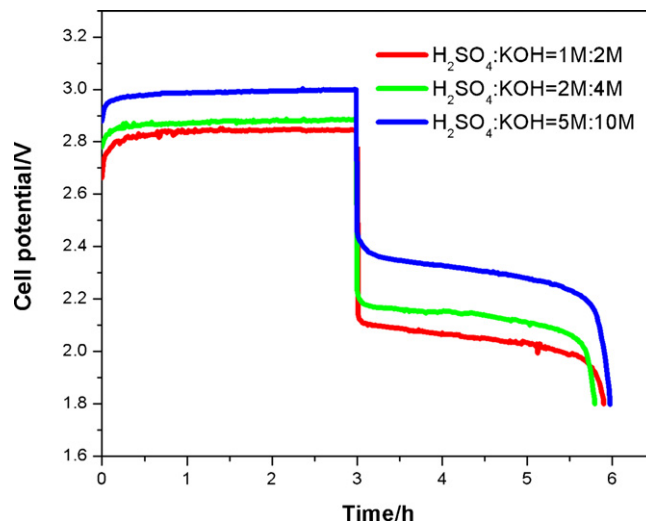


Fig. 6. Discharge performance of hybrid cell at different acid–alkaline concentration. The discharging current was 100 mA and the salt solution in the middle chamber was 0.2 M K_2SO_4 .

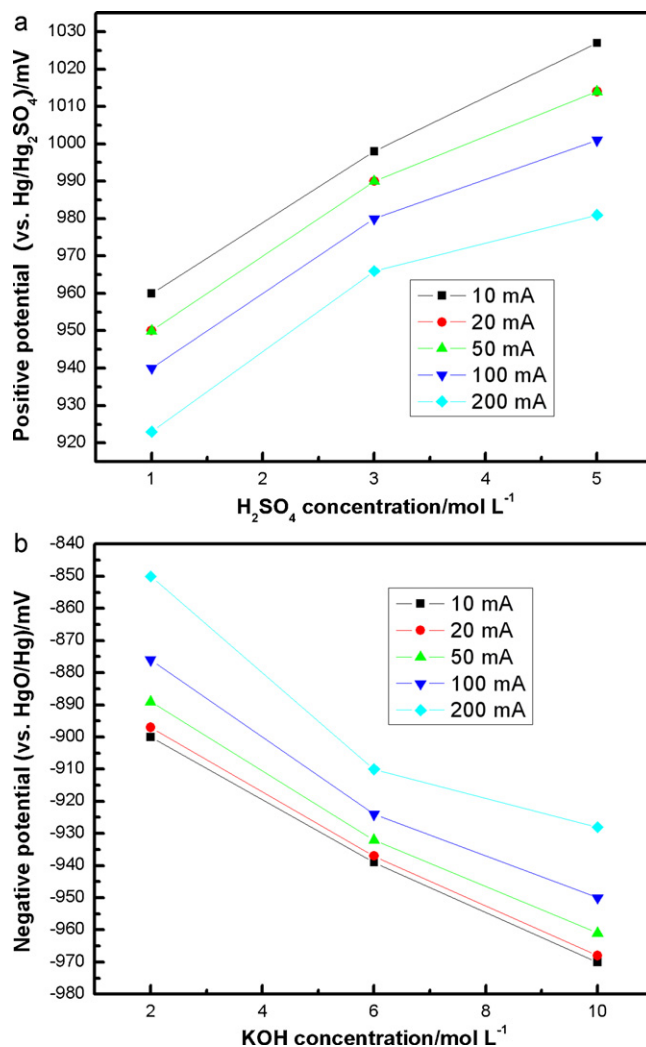


Fig. 7. Potential values of positive (a) and negative (b) electrodes in different concentrations of acid and alkaline at different discharge rates. The salt solution in the middle chamber was maintained at 0.2 M K_2SO_4 with 0.5 cm gap.

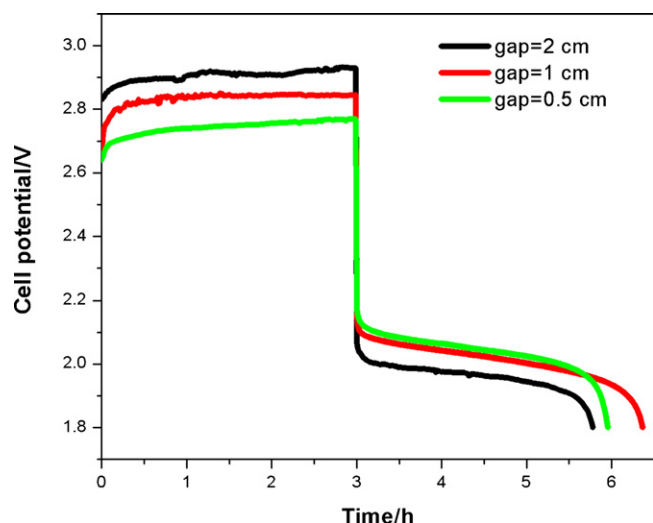


Fig. 8. The charge/discharge curve of 1 M H₂SO₄/0.2 M K₂SO₄/2 M KOH hybrid cell with various width of the central compartment (current = 100 mA).

the membranes and increase possibility of SO₄²⁻ and K⁺ ions crossing over, resulting in salt formation.

3.5. Effect of varying the distance between the acid and alkaline chambers

We varied the distance between the acid and alkaline chambers from 0.5, 1.0 to 2.0 cm. The hybrid system is able to maintain its discharging time close to 3 h for all distances. However, as the distance increased, the charging plateau voltage increased from 2.7 V for 0.5 cm gap to 2.8 and 2.9 V for 1 cm and 2 cm gap respectively, as shown in Fig. 8.

3.6. Theoretical specific energy/energy density of the three-electrolyte cell

The specific energy of a battery cell can be calculated as specific capacity of the pair of electrodes $\times$ operating voltage,

$$\frac{C_1 C_2}{C_1 + C_2} \times \text{operating voltage} \quad (11)$$

where C_1 and C_2 are the specific capacity of individual positive and negative electrodes.

The theoretical specific capacities of LaNi₅-based materials is 340 mAh/g [23] and lead oxide is 224 mAh/g [24]. The theoretical specific energy of the individual nickel metal hydride battery and lead acid battery are calculated to be 187 Wh/kg and 240 Wh/kg, respectively, assuming operating voltages of 1.2 V and 2.0 V respectively. Assuming the electrolyte and other components do not contribute to the mass, the theoretical specific capacity of the 3-electrolyte battery system with the MH–lead acid pair is calculated to be 135 mAh/g (135 Ah/kg). As a result, the theoretical specific energy of the 3-electrolyte battery system is 135 Ah/kg $\times$ 2.6 V = 351 Wh/kg. Indeed, the theoretical specific energy of the 3-electrolyte is higher than the individual batteries, such improvement is due to the pH difference, as well as the free energy from electrochemical neutralization.

In practice, considering the 3-electrolyte system with a 0.5 cm gap (as observed from Fig. 8), the system can be discharged for 2.97 h at 100 mA and the average operating discharge voltage is 2.04 V. Thus the best estimate of the present experienced practical specific energy of this 3-electrolyte system is 202 Wh/kg. The

difference in specific energy between the theoretical and practical values can be attributed to (1) electrolyte mass, (2) internal resistance (across ionic interfaces as in Eq. (9)), and (3) internal resistance through electrolyte. All these will depend on magnitude of current and affect the performance of the battery system. In order to improve the performance of the system in future, we expect to reduce the electrolyte mass by compacting the size of the acidic and alkaline chambers.

4. Conclusions

An acid–alkaline hybrid battery with an alternative ionic interfaces was proposed in this paper. The open circuit voltage of this hybrid cell was found to be as high as 2.64 V in 1 M H₂SO₄–0.2 M K₂SO₄–2 M KOH electrolytes, compared to 1.92 V in a conventional lead–acid cell in 1 M H₂SO₄ and 1.40 V of the NiMH_x cell in a 2 M KOH electrolyte. Under a 1/3 C discharge rate, this hybrid acid–alkaline PbO₂/NiMH_x battery had shown to operate with a voltage 20% higher than the conventional lead acid battery and 110% higher than the conventional nickel–metal hydride battery. The choice and concentration of the salt solution in the middle compartment, as well as the concentration of these three electrolytes, which affects the internal resistance of the cell and polarization of positive and negative electrode, are the main factors that have been identified to have an impact on overall performance of this hybrid battery.

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